

X-RAY POWDER DIFFRACTION (XPD)

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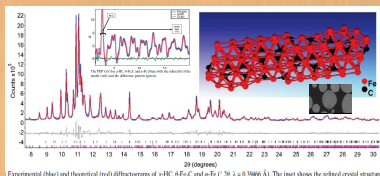
TECHNIQUES AND CAPABILITIES

- **Techniques:** powder diffraction, total scattering (PDF), small/medium angle scattering (SAXS, MAXS), tomography (CT). Opportunities for *in-situ* and *in-operando* structural science (variety of sample environments, functional materials in working conditions or in operating devices).
- **Source:** 1.8 T Damping Wiggler, $E_c = 10.8$ keV.
- **X-ray Energy Range:** 30-70 keV
- **Energy Resolution:** 10^{-3} (high flux configuration) to 2×10^{-4} $\Delta E/E$ (high resolution configuration)
- **Spatial Resolution:** ~mm horizontal beam size (powder averaging) down to ~10 μm
- **Time Resolution:** sub-sec (dynamics, rapid acquisition)
- **Beamline components:** cryo-cooled Laue monochromator, vertically focusing mirror (Fig. 2), channel-cut high resolution monochromator.

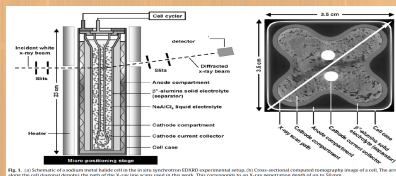


XPD features two independent branches (XPD1 and XPD2 – PDF) served by Laue-crystal monochromators and three in-line endstations fitted with high-resolution, position-sensitive and large-area detectors. XPD is capable of collecting data at high energies ideal for high-Q data and *in situ* and time-resolved studies in environmental cells.

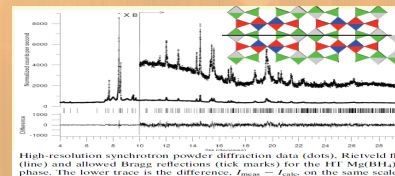
APPLICATIONS



High energy total structure: XPD offers to examine materials *in situ* over the mm-nm-Å length-scales by combining high Q-space resolution (long-range crystallographic structure) and high real-space resolution (Pair Distribution Function): local structure or nanostructure). The example above is a combined Rietveld and PDF study of Fe_3C_2 carbide from H. Esna du Plessis *et al.* J. of Synchrotron Rad. 18 (2011). Quantitative Rietveld and PDF measurements reveal the structure in the core and over the surface of the active Fe_3C_2 catalyst nanoparticles.



Space and time resolved high energy diffraction: XPD is optimized for *in operando* time and space studies of the electrochemical processes taking place during charge/discharge cycling at high temperature inside a full size battery cell. The example above is a prototype sodium metal halide cell from J. Rijnssenbeek *et al.* J. of Power Sources 196 (2011). XPD will aid the advancement of battery technology by enabling the study of the fundamental mechanisms at work inside commercially relevant cells of different battery types.



In situ high energy diffraction: The structure of H_2 storage material is to date determined *ex situ* on recovered materials. XPD aims instead at studying the material *in operando* under high temperature and H_2 pressure gradients with sub-second time resolution (intermediate states are examined during the decomposition process). Knowledge of the structure is fundamental to help understand hydrogen desorption and find a practical means of regenerating the material. The example is from Jae-Hyuk Her *et al.* Acta Cryst. B63 (2007).

RESEARCH & DEVELOPMENT PROJECTS



Fig. 1: view of the XPD hutches (First Optical Enclosure; XPD2 endstation; XPD1 endstation)



Fig. 2: view of the XPD mirror during the polishing process at the factory

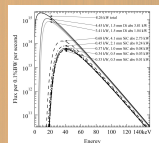


Fig. 3

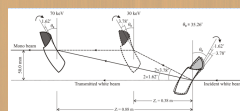


Fig. 4

- **High-energy bandpass filtering:** the power of the wiggler source (61 kW) is reduced by the means of 2 fixed-aperture masks and 2 fixed, redundant diamond windows followed by a set of moveable SiC filters. The diamond windows serve as heat filter, shield and vacuum barrier. Aggressive filtering causes beam hardening. Fig. 3 shows the power spectrum distribution.
- **Double Laue Monochromator (DLM):** the sagittal bending of the asymmetric Laue crystals focuses the incident horizontal fan (35 mm to 0.5 mm). Severe crystal distortions broaden the energy bandwidth and produce a significant gain in flux. Efficient crystal cooling and control of the sagittal and the anticlastic bends are challenging (X. Shi *et al.* J. Appl. Cryst. 2011). This DLM will be a unique instrument acting as the first white-beam optics (Fig. 4).
- **Laue analyzer optics:** the high-resolution mode of XPD uses a post-sample array of parallel crystals. All existing powder diffractometer designs rely on the concept developed at the CNRS-ESRF by J.-L. Hodeau *et al.* and operate below 40 keV with Bragg crystals. The proposed solution consists of an assembly of parallel Laue crystals in a pseudo-Rowland circle geometry and combined with a multi-channel detector.
- **High energy detectors:** the low absorption of high-energy x-rays by silicon drastically limits the efficiency of Si-based detector technology. Pending successful engineering of higher-Z sensors in the coming years, a 10-fold increase in detector efficiency at 60 keV is expected. A multi-stripe photon-counting CdTe detector is being considered.